CERTIFICATION REPORT

The certification of absorbed energy (150 J nominal) of a Master Batch of Charpy V-notch reference test pieces:

Certified Reference Material ERM®-FA415s

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The certification of absorbed energy (150 J nominal) of a Master Batch of Charpy V-notch reference test pieces:

Certified Reference Material ERM®-FA415s

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Summary

This certification report describes the certification of ERM®-FA415s, a batch of steel Charpy V-notch certified reference test pieces. This batch will serve as a Master Batch, to be used by IRMM for the certification of secondary batches. Each CRM unit consists of a set of five test pieces taken from a secondary batch are distributed by IRMM and its authorised distributors for the verification of pendulum impact test machines according to ISO 148-2 [1].

The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1 [2]. The certified value for *KV* is an estimate of the mean value of the whole batch. The obtained values are shown in the table below. The associated uncertainties are standard uncertainties corresponding to a confidence level of about 68 %. The certified value is traceable to the International System of Units (SI). The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

STEEL CHARPY V-NOTCH TEST PIECES		
	Impact toughness	
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	157.3	1.4

1) The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in ISO 148-1 [2]. The certified value is only valid for strikers with a 2 mm tip radius and in the temperature range of (20 ± 2)°C.

2) The certified value is estimated as the mean of means of absorbed energies measured on 7 pendulums at 5 different laboratories. On each pendulum, 20 test pieces were broken. The pendulums are regularly verified with equipment that is calibrated in a manner that is traceable to the International System of Units (SI). Therefore, the certified value is traceable to the International System of Units (SI).

3) Standard uncertainty *u* of the certified mean absorbed energy of batch ERM-FA415s, estimated as the standard error of the mean of the 7 pendulum mean values, corresponding with a confidence level of about 68 %, as defined in the ISO/IEC Guide 98-3:2008; Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM) [3]. The number of degrees of freedom of the certified uncertainty, $\nu_{RM} = 6$.

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Glossary

AISI	American Iron and Steel Institute
ASTM	now ASTM international, formerly American Society for Testing and Materials
BCR	Community Bureau of Reference
BELAC	Belgische Accreditatie-instelling
CRM	Certified Reference Material
ERM®	Trademark of the European Reference Materials
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements of the JRC
ISO	International Organization for Standardization
JRC	Joint Research Centre
KV	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
KV_{char}	Mean of pendulum mean KV values
KV_{CRM}	Certified KV value of a unit of 5 reference test pieces from the Secondary Batch
KV_{MB}	Certified KV value of the Master Batch test pieces
LNE	Laboratoire national de métrologie et d'essais
MB	Master Batch
p	Number of accepted data sets in the characterisation study
RSD	Relative standard deviation
s	Standard deviation
SB	Secondary Batch
s_{char}	Standard deviation of the mean values of the accepted data sets in the characterisation study
s_{hom}	Standard deviation of the results of the test pieces tested to assess the homogeneity of the Master Batch
SI	International System of Units

s_{pen}	Standard error of a mean value of a data set from the characterisation study
s_{SB}	Standard deviation of the results of the test pieces tested to assess the homogeneity of the Secondary Batch
s_{within}	Standard deviation of a data set from the characterisation study
T	Temperature
u	Standard uncertainty
u_{char}	Standard uncertainty of the result of the characterisation tests
$u_{\text{hom,MB}}$	Contribution to uncertainty of the MB certified value from homogeneity
$u_{\text{hom,SB}}$	Contribution to uncertainty of the SB certified value from homogeneity
u_{lts}	Standard uncertainty derived from a long term stability study
u_{MB}	Combined standard uncertainty of KV_{MB}
u_{sts}	Standard uncertainty derived from a short term stability study
W_{t}	Total absorbed impact energy as measured with an instrumented Charpy pendulum
$\overline{X}_{\text{MB}}$	Mean KV value of the n_{MB} measurements on test pieces of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{\text{SB}}$	Mean KV value of the n_{SB} results of the test pieces tested for the characterisation of the Secondary Batch
Δh	difference between the height of the centre of gravity of the hammer prior to release and at the end of the half-swing during which the test piece is broken
ν_{char}	Number of degrees of freedom associated with the uncertainty of the characterisation study
ν_{RM}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1 Introduction

1.1 Background: the Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

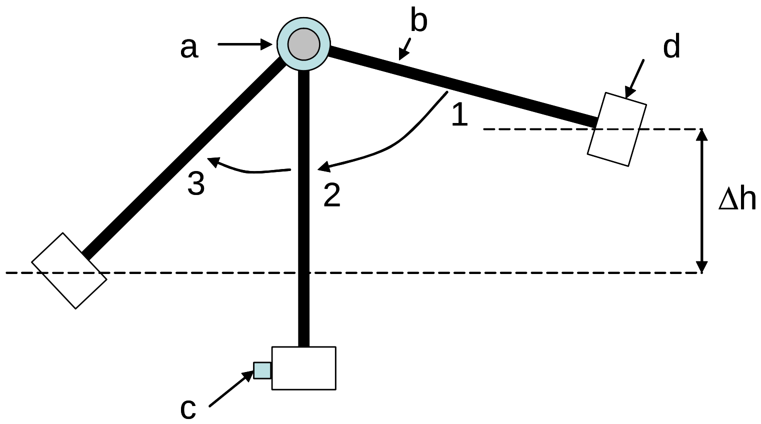

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which the hammer is fixed, d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test piece, which is positioned on a support and against the pendulum anvils (not shown). The height reached by the hammer after having broken the test piece (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds with a difference in potential energy, and is a measure of the energy required to break the test piece.

The energy absorbed by the test piece is very dependent on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in ISO and other international standards [1,4]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [5], schematically shown in Figure 2.

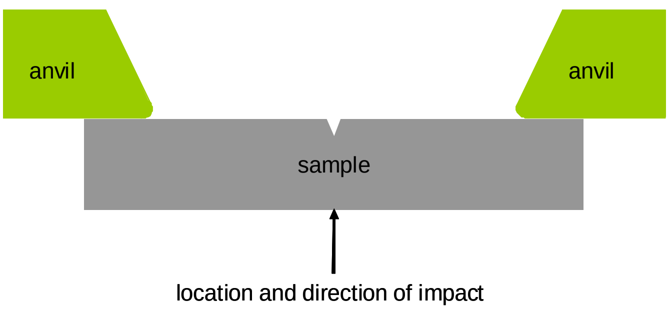

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact.

1.2 Design of the project: the certification concept of Master Batch and Secondary Batch

1.2.1 Difference between Master and Secondary Batches

This report describes the production of a “Master Batch” (MB) of Charpy V-notch certified reference test pieces. This work was performed in accordance with procedures described in the BCR reports [6] and [7], and in compliance with the ISO Guide 34 [8] requirements for the producers of certified reference materials (CRMs). IRMM is accredited by BELAC for the production of Charpy reference materials according to ISO Guide 34. The certified value of a master batch is obtained using an international interlaboratory comparison, in accordance with ISO Guide 34.

The certification of a secondary batch (SB) is based on the comparison of a unit of SB test pieces with a unit of MB test pieces having a similar absorbed energy, using a single pendulum under repeatability conditions. The BCR reports [6] and [7] describing the SB certification approach, were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO Guide 98 (Guide to Expression of Uncertainty in Measurement [3]). This revised approach was developed and presented by Ingelbrecht *et al.* [9,10], and is summarised below.

1.2.2 Certification of a Secondary Batch of Charpy test pieces

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean *KV*-value of a unit of SB-test pieces ($\overline{X}_{\mathrm{SB}}$) tested on a single pendulum. This value $\overline{X}_{\mathrm{SB}}$ has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the test pieces of the SB, is estimated by comparing the mean *KV*-value of a number of test pieces of the MB ($\overline{X}_{\mathrm{MB}}$), tested together with the SB test pieces under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows:

$$KV_{CRM} = \left[\frac{KV_{MB}}{\overline{X}_{MB}} \cdot \overline{X}_{SB} \right] \qquad \text{Equation 1}$$

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB test pieces is required. This is the reason why MB and SB test pieces are made from nominally the same steel.

1.2.3 Uncertainty of the certified value of a Secondary Batch of Charpy test pieces

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Eq. 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio $\overline{X}_{\mathrm{MB}} / \overline{X}_{\mathrm{SB}}$. The full measurement uncertainty of the values $\overline{X}_{\mathrm{MB}}$ and $\overline{X}_{\mathrm{SB}}$ is relatively large. However, when all conditions mentioned above (repeatability conditions, pendulum performance, and commutability between Secondary and Master Batch) are fulfilled, then the uncertainties of the values $\overline{x}_{\mathrm{MB}}$ and $\overline{x}_{\mathrm{SB}}$ have several contributions in common, in particular the uncertainty due to the bias of the pendulum. These shared uncertainty components do not contribute to the uncertainty of the ratio $\overline{x}_{\mathrm{MB}} / \overline{x}_{\mathrm{SB}}$, and only the standard deviations of the SB and MB results in the MB-SB comparison test need to be taken into account.

1.2.4 Implications for the required uncertainty of the certified value of a Master Batch of Charpy test pieces

Implementing the MB-SB approach has an important implication for the nature of the value that is required for the factor KV_{MB} in Eq.1. Indeed, the certified value and associated uncertainty that have to be determined for a Master Batch are the best estimate of the average of all test pieces in the Master Batch, and the uncertainty of this average value. This means that the test piece-to-test piece heterogeneity, which traditionally is an essential contribution to the uncertainty of the certified property value of a reference material, will not be included in the uncertainty of the certified value of the Charpy Master Batches. Instead, the heterogeneity of the Master Batch will be included in the uncertainty of the certified value of a future Secondary Batch via the uncertainty of the factor $\overline{x}_{\mathrm{MB}}$ in Eq.1.

2 Participants

2.1 Project management and evaluation

- European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE
  (accredited to ISO Guide 34 for production of certified reference materials, BELAC No 268-RM)

2.2 Processing

- Cogne Acciai Speciali, Aosta, IT: production of steel bars
- Aubert & Duval, Gennevilliers, FR: heat treatment of steel bars
- Laboratoire National de Métrologie et d'Essais (LNE), Trappes, FR: processing of the V-notch test pieces

2.3 Homogeneity study

- European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE
  (accredited to ISO Guide 34 for production of certified reference materials, BELAC No 268-RM; measurements under the scope of ISO/IEC 17025 accreditation BELAC 268-Test)

2.4 Stability study

- European Commission, Joint Research Centre, Institute for Reference Materials and Measurements (IRMM), Geel, BE
  (accredited to ISO Guide 34 for production of certified reference materials, BELAC No 268-RM; measurements under the scope of ISO/IEC 17025 accreditation BELAC 268-Test)
- Laboratoire National de Métrologie et d'Essais (LNE), Trappes, FR
  (measurements under the scope of ISO/IEC 17025 accreditation COFRAC SMH 2-1287)

2.5 Characterisation

The following laboratories participated in the interlaboratory characterisation:

Non-instrumented tests:

- Exova, Emmen, NL
  (measurements under the scope of ISO/IEC 17025 accreditation, RvA testen L085)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Division 9.1 Random Loading Fatigue and Structural Integrity, Berlin, DE
  (measurements under the scope of ISO/IEC 17025 accreditation, DGA-PL-2614.16)
- Centro de Apoio Tecnologico a Industria Metalomechanica (CATIM), Laboratório de Ensaios, Porto, PT
  (measurements under the scope of ISO/IEC 17025 accreditation, IPAC L009)
- Fraunhofer-Institut für Werkstoffmechanik IWM, Freiburg, DE
- European Commission Joint Research Centre (JRC), Institute for Reference Materials and Measurements, Geel, BE
  (measurements under the scope of ISO/IEC 17025 accreditation BELAC 268-Test)
- Laboratoire National de Métrologie et d'Essais, Charpy Laboratory, Trappes, FR
  (measurements under the scope of ISO/IEC 17025 accreditation COFRAC SMH 2-1287)
- National Institute of Standards and Technology (NIST), Materials Reliability Division, Boulder, USA
- SCK-CEN, Labo Reactormaterialenonderzoek, Mol, BE
  (measurements under the scope of ISO/IEC 17025 accreditation BELAC 015-Test)
- SIRRIS, Beproevingslaboratorium Gent, Zwijnaarde, BE
  (measurements under the scope of ISO/IEC 17025 accreditation BELAC 232-Test)
- Universität Stuttgart, Materialprüfungsanstalt, Stuttgart, DE
  (measurements under the scope of ISO/IEC 17025 accreditation DAP-PL-2907.02)

Instrumented tests:

- Bundesanstalt für Materialforschung und -prüfung (BAM), Division 9.1 Random Loading Fatigue and Structural Integrity, Berlin, DE
  (measurements under the scope of ISO/IEC 17025 accreditation, DGA-PL-2614.16)
- SCK-CEN, Labo Reactormaterialenonderzoek, Mol, BE
  (measurements under the scope of ISO/IEC 17025 accreditation BELAC 015-Test)
- Fraunhofer-Institut für Werkstoffmechanik IWM, Freiburg, DE

3 Material processing and process control

The processing of the steel test pieces consisted of the following main steps:

- Melting and casting of a steel ingot with appropriate composition, and subdivision of the ingot into a number of smaller billets.
- Hot-rolling of the billets into long (4 to 7 m) bars of square cross-section (about 12 mm x 12 mm).
- Heat treatment of the bars to obtain the appropriate steel microstructure.
- Cutting of the bars into pieces, and machining of rectangular test pieces (55 mm x 10 mm x 10 mm).
- Machining a V-notch in each test piece.

3.1 From steel to hot-rolled bars

The base material consisted of ASTM 565 XM-32 steel [12]. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the compositional tolerances specified in Table 1 were imposed on the selected steel batch. These tolerances are stricter than generally allowed for ASTM 565 steel.

Table 1: Adapted composition tolerances of ASTM 565 Grade XM-32 [12]

Composition (mass %)						
C	S	P	Si	Mn	Cr	Ni
0.11 - 0.13	< 0.003	<0.018	0.15 – 0.3	0.75 – 0.9	11.25 – 11.65	2.55 – 2.75
Mo	Cu	Al	V	W	N	
1.55 – 1.7	< 0.2	< 0.01	0.25 – 0.3	< 0.1	0.025 - 0.04	

The ingot was prepared and hot rolled at Cogne Acciai Speciali (Aosta, IT), resulting in bars that were 4 m to 7 m long and with a square cross-section of 12 mm × 12 mm. For the ERM®- FA415s batch, steel was used from ingot number 360404. The length of the selected bars was approximately 4.5 m. A full description of the processing and quality check of the steel bars is available in [13].

3.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert & Duval, Gennevilliers (FR), under the conditions indicated in Table 2.

Table 2: Heat treatment conditions

Batch	Number of bars	Austenisation		Annealing	
		T (°C)	Time (min)	T (°C)	Time (min)
ERM®-FA415s	20	980	31	745	300

During the heat treatment, bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C'.[1] From this furnace, the bars were quenched in oil at 40 °C. After the oil-quench, the test pieces were annealed in a second furnace ('class 5 °C'). After this annealing treatment, the test pieces were cooled down in ambient air.

3.3 Machining of Charpy test pieces

After the heat treatment, test pieces were machined from the bars to dimensional tolerances imposed in ISO 148-3 [5].[2] In this production step, the major part of the microstructural gradient from test piece surface to test piece core is removed. The batch code ('S 160') was engraved on one of the two end faces of the test piece.

The V-notch was introduced using electric discharge machining. Since the notch is 2 mm deep, its tip is well below the surface layer, the properties of which might be affected to some extent by the near-surface gradient in microstructure resulting from the successive heat treatments.

[1] In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

[2] During the certification project, the ISO 148 standards have been updated. None of the geometrical test piece characteristics have been affected in a manner that would disqualify the test pieces. In any case, the internal specifications of the processing subcontractor are stricter than the ISO 148 criteria.

Both machining and notching operations are performed in accordance with strict and controlled procedures.

3.4 Quality control

Once all test pieces from the batch were fully machined, a randomised selection of 25 test pieces was made. The dimensions of the 25 test pieces were checked on November 17, 2003. They all meet the tolerances specified in ISO 148-3 [5]: length $55.00^{+0.00}_{-0.30}$ mm, height (10.00 ± 0.06) mm, width (10.00 ± 0.07) mm, notch angle (45.0 ± 1.0) °, height remaining at notch root (8.00 ± 0.06) mm, radius at notch root (0.250 ± 0.025) mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece (27.5 ± 0.2) mm. All test pieces met all requirements.

The test pieces checked for geometrical compliance were tested on November 18, 2003 on the Tinius Olsen 350 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificate LNE No. C111076/CQPE/5 [14]. The average *KV* of the 25 test pieces, at room temperature, was 157.9 J, sufficiently close to the target value (150 J). The standard deviation of the test results (s = 4.7 J, *RSD* = 3.0 %) was smaller than the maximum level of 5 % allowed by ISO 148-3 [5]. An additional 25 test pieces were impact tested on December 4, 2003 on the same pendulum at LNE. The results are reported in certificate LNE No. C111076/CQPE/6 [15]. The average *KV* of the 25 test pieces, at room temperature, was 158.7 J, and the standard deviation of the test results was s = 4.1 J (*RSD* = 2.6 %). The test piece-to-test piece homogeneity was checked again during the characterisation tests (see also section 4).

3.5 Packaging and storage

The test pieces were packed in oil-filled and closed plastic bags in sets of 5. The test pieces were randomly picked to ensure a mixture of test pieces from 5 different bars in each unit. The test pieces were closely packed in the bag to eliminate the possibility that corners or edges of one bar scratch the other bars. The oil-filled bags, together with a label, were packed in a sealed plastic bag, and shipped to IRMM. The 250 units of ERM-FA415s test pieces (delivery January, 2004) were registered and stored at room temperature.

4 Homogeneity

4.1 Between-test piece homogeneity

The homogeneity of the ERM-FA415s batch was assessed at IRMM on February 24 and 25, 2009, when 10 test pieces were tested on each day, with *RSD* values of 1.5 % and 2.1 % respectively. The higher value of both results is chosen as a conservative estimate of s_{hom}, the test piece-to-test piece heterogeneity of ERM-FA415s. This value meets the homogeneity criterion imposed by ISO 148-3 [5] on batches of certified reference test pieces for Charpy impact tests (*RSD* < 5 %).

4.2 Homogeneity contribution to the uncertainty of KV_{MB}

The certified value of ERM-FA415s pertains to the average of the whole batch (see also section 1.2.4). Therefore, one has to investigate how the test piece-to-test piece heterogeneity may affect the estimation of the average value of the batch. This certified value is determined as the mean of the pendulum mean values collected in an interlaboratory study (see section 6). Since 20 test pieces were tested per pendulum, the standard error of each of the pendulum mean values can be deduced from s_{within}, the within-pendulum standard deviation, as in Eq. 2.

$$s_{\text{pen}} = \frac{s_{\text{within}}}{\sqrt{20}} \qquad \textbf{Equation 2}$$

Assuming that all pendulums reveal a comparable s_{within}, the effect of the corresponding s_{pen} on the uncertainty of KV_{MB} depends on the number of participating pendulums, *p:*

$$u_{\text{hom,MB}} = \frac{s_{\text{pen}}}{\sqrt{p-1}} \qquad \textbf{Equation 3}$$

The 20 test pieces tested on each pendulum were selected from the batch of about 1250 test pieces. Test pieces were randomly selected from all bars constituting the batch, and from all positions along the bars. Choosing the value of s_{hom} (2.1 %, or 3.3 J; see section 4.1) as a representative value for s_{within}, the within-pendulum standard deviation due to test piece-to-test piece heterogeneity, s_{pen} , is 0.74 J, and the value of $u_{\text{hom,MB}}$ calculated with Equation 3, is 0.3 J.

5 Stability

Microstructural stability of the certified reference test pieces is obtained by the annealing treatment to which the test pieces were subjected after the austenisation treatment[3]. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature (α-Fe and Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α-Fe matrix between the annealing and the ambient or storage temperatures. Relaxation of residual (micro-)stresses by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly.

Given the test piece-to-test piece heterogeneity of about 2 % (see section 4.1), the ageing effects are difficult to detect when testing limited numbers of test pieces. Extensive efforts have been spent to quantify the stability of the certified values of batches of Charpy CRMs. The first systematic investigation was performed for test pieces of nominally 120 J by Pauwels *et al.*, who did not observe measurable *KV* changes over a period of 1.5 years, even with exposure to 90 °C [16]. New evidence for the stability of the reference test pieces produced from AISI 4340 steel of other energy levels (nominally 15 J, 30 J and 100 J) has been obtained during the International master batch (IMB) project [18]. In the IMB-project, the stability of the certified test pieces is

[3] Austenisation is the treatment whereby the steel is brought to a temperature inside the range where the austenite phase (γ-Fe) is stable.

confirmed by the unchanged value of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period.

A confirmation of the stability of the *KV* value of ERM-FA415s was obtained by comparing results obtained over time at IRMM and LNE, where 9 series of tests were performed between 18/11/2003 and 9/2/2012, on ERM-FA415s test pieces which during this 100 months period were stored at room temperature. To make a single stability assessment, the data obtained on both pendulums were pooled by normalising to the average value of the earliest data set obtained on the respective pendulum. This normalisation should remove the small but consistent differences between the IRMM and LNE pendulums. Figure 3 confirms that the *KV* values are stable: the very small slope of -0.00004 %/month is not significant (at 99 % confidence level).

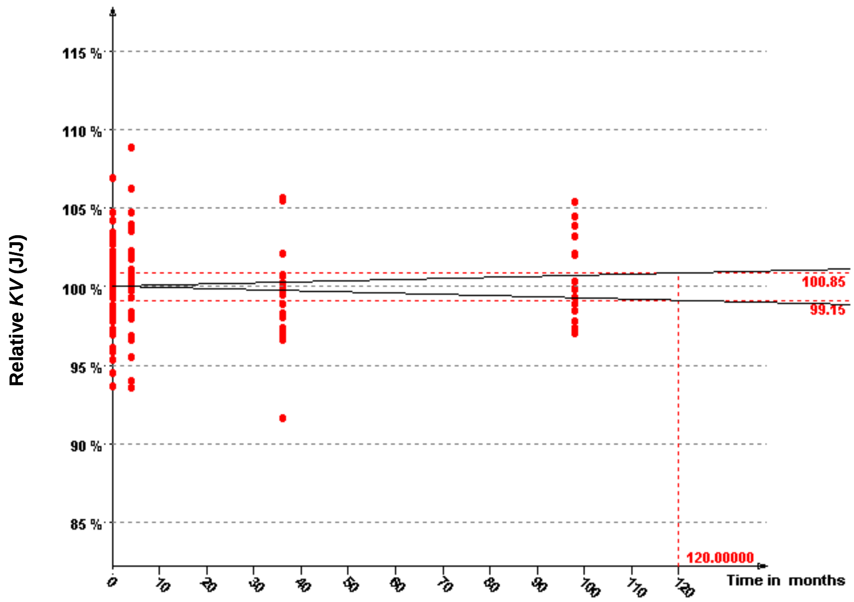

Figure 3: Evaluation of the stability of the KV value of ERM-FA415s based on results obtained at LNE and IRMM between 2003 and 2012. Individual data points represent results obtained on ERM-FA415s test pieces at LNE and IRMM (relative values [J/J]). The wedge-like double curve indicates the possible variation of the average KV value with time. The indicated values at the right of the graph indicate lower and higher margins (in % J/J) of the uncertainty interval associated with stability under 10 years of storage at ambient temperature.

The standard uncertainty of the *KV* value of ERM-FA415s, u_{lts}, that would be derived from the data in Figure 3 is 0.85 %, or about 1.3 J, for a storage period of 120 months at room temperature (18 °C). This uncertainty is judged to be due to the scatter in data at the individual time points, and less to the stability of the material. It is therefore decided not to add an uncertainty contribution from instability in the combined uncertainty of the certified value.

Taking into account the above, it is decided to specify a shelf-life of 10 years, counting from the date of the characterisation tests. Since the materials were characterised between December 2011 and February 2012, the validity of the ERM-FA415s certificate stretches until December, 2021. The proposed shelf-life may be extended as further evidence of stability becomes available. An isochronous post-certification monitoring test has been initiated in April 2012.

Since it is believed that the steel is stable even at 60 °C, this temperature was chosen as the test temperature in the stability study (reference temperature 18 °C, time-points 15, 30, 45 and 60 months). Results will be available in May 2017.

6 Characterisation

6.1 Selection of participants

Characterisation of the candidate master batch was carried out in an interlaboratory comparison between a statistically representative set of 12 pendulum impact testing machines (P1 to P12, from 10 different laboratories). 5 of the pendulum hammers were of the U-type, and 7 of the C-type. The instrument's maximum loading capacity was not used as a selection criterion. 3 laboratories performed instrumented Charpy tests, the results of which can provide additional information to better understand the results of the non-instrumented tests.

The laboratory selection was a multi-step process. First a list of laboratories with sound expertise and demonstrated competence in the field of 'mechanical testing' was put together based on an open call for interest. Laboratories were selected on the basis of a combination of quality management and technical criteria. All laboratories have a quality system, and most of them are accredited to ISO/IEC 17025 [11]. Results of proficiency testing schemes or other published interlaboratory comparison data were used to assess the technical quality of the laboratories. In summer 2011, laboratories qualified for the 'mechanical testing' field, were invited to submit a tender for the execution of Charpy impact tests in accordance with ISO 148-2 [1]. A further selection was then made based on an evaluation of these tenders in terms of cost, and in terms of technical criteria specific for Charpy impact tests, such as the control over anvil spacing and temperature, the results from direct verification tests control (hammer tip and anvil radii) and results from tests on reference materials.

Details of the 12 pendulums used in this study are given in Annex 1. All selected pendulums are regularly verified with instruments and tools that are traceable to the respective national standards. This is essential, because it implies that the measured values, as well as the resulting certified value of the master batch, will be traceable to the SI.

The interlaboratory comparison exercise was performed between December 2011 and February 2012.

6.2 Study setup

On each pendulum, 20 test pieces of the ERM-FA415s batch were tested, corresponding to 4 units of 5 test pieces randomly selected from the whole batch. A strict test protocol was imposed, referring to the ISO 148 series of standards [1, 2, 5], including the correction of the raw data for friction, and additionally imposing a randomised order of the tests, distributed over two test days (10 test pieces on day 1, 10 test pieces on day 2). All tests were to be performed at (20 ± 2) °C.

For quality control purposes, the test protocol also included the testing of 10 test pieces (5 test pieces on day 1 and 5 test pieces on day 2) of a secondary batch CRM (FA415v), which was not yet released at the moment of the FA415s interlaboratory characterisation study. Therefore, on both testing days, 15 test pieces had to be tested. The order of testing the test pieces was fully randomised, mixing the different batches. The average values of the FA415s and FA415v batches were expected to be slightly different, due to small differences in the material's microstructure.

The laboratories performing instrumented impact tests were requested to follow the testing and reporting procedures described in ISO 14556:2000 [18]. The test schedule was the same as for the non-instrumented tests. Actually, all three laboratories obtained their instrumented and non-instrumented data simultaneously, on the same test pieces: when testing a test piece, the traditional data (from the measurement of the angle of rise of the pendulum, by an electronic inclinometer or by a pointer) and the 'instrumented' data (from the strain gauges on the hammer tup) can be recorded simultaneously.

6.3 Evaluation of results

6.3.1 Technical evaluation

The reporting laboratories successfully tested all test pieces, and reported all corresponding data points. IRMM collected all broken test pieces.

Compliance with measurement protocol

For all pendulums, except P6, the tolerance of ± 2 °C was respected. In fact, most laboratories provided a test piece temperature control better than ± 1 °C, as this was one of the tender award criteria. The data of P6, which did not respect the required test temperature, were discarded.

A Grubbs' statistical outlier test was performed on the data for each individual pendulum (except on the discarded data of P6), both on the ERM-FA415s and on the ERM-FA415v data, which are shown in Annex 2. No outlier values (99 % confidence level) were detected with the Single and Double Grubbs' tests. All data obtained on an individual pendulum were normally distributed.

No analytical trends were observed except for P11 (for both FA415s and FA415v data). The trends were significant at the 99% confidence level and were very pronounced. The slopes (visible in the corresponding graph in Annex 2) are due to a significant difference between the results obtained on the two test days. This indicates that the testing conditions were not stable. Closer inspection of the pendulum indicated a crack in the pendulum base. The data of P11 were therefore discarded.

Compliance with requirements of ISO 148-3 for reference pendulums

For each pendulum the average value obtained on the 10 test pieces of CRM FA415v test was compared with the bias criterion taken from ISO 148-3 [5], which states that the difference between the average value obtained on the pendulum shall not deviate from the certified value by more than 5 %. The mean values from P6 (+11 %), P7 (+12 %), P9 (+10 %), P11 (+11 %) and P12

(+6 %) are higher than the upper 5 % range. Therefore, the data of these pendulums had to be eliminated from further analysis.

The ISO 148-3 [5] repeatability criterion requires reference pendulums to produce a standard deviation < 7.5 % for results obtained on at least 10 reference test pieces. Based on the 10 results obtained on FA415v samples, over 2 test days, this criterion was not met by pendulums P6 (8.6 %), P7 (9.8 %), P8 (8.3 %), P9 (8.2 %) and P11 (10.7 %). However, since the tests were performed over 2 different days, this does not correspond with tests in repeatability conditions. A second, more robust standard deviation was therefore calculated, based on the 20 FA415s test pieces. Most laboratories obtained a lower standard deviation, and only pendulum P9 (7.9 %) failed the test. Since the results of P9 were already eliminated based on the bias criterion, applying the repeatability criterion did not result in the elimination of additional data sets.

Discussion of results of technical evaluation

It is noted that for all eliminated pendulums, the measured values were too high. There is some logic in this observation, in the sense that the vast majority of pendulum problems will lead to a loss of energy, rather than making the test piece break more easily. A second observation is that most eliminated data (i.e. P7, P9 and P12) were obtained on pendulums for which the most recent annual direct verification (the results of which had to be reported in the laboratory selection process) were performed at lower *KV* levels than the *KV* range of the ERM-FA415 batches. P6 and P11 were verified at a relevant *KV* level, but the corresponding data were affected by problems mentioned earlier (P6: test temperature; P11: analytical trend).

6.3.2 Statistical evaluation of the accepted results

Average values of the accepted data sets obtained on batch ERM-FA415s (20 test pieces/pendulum) are shown in Figure 4.

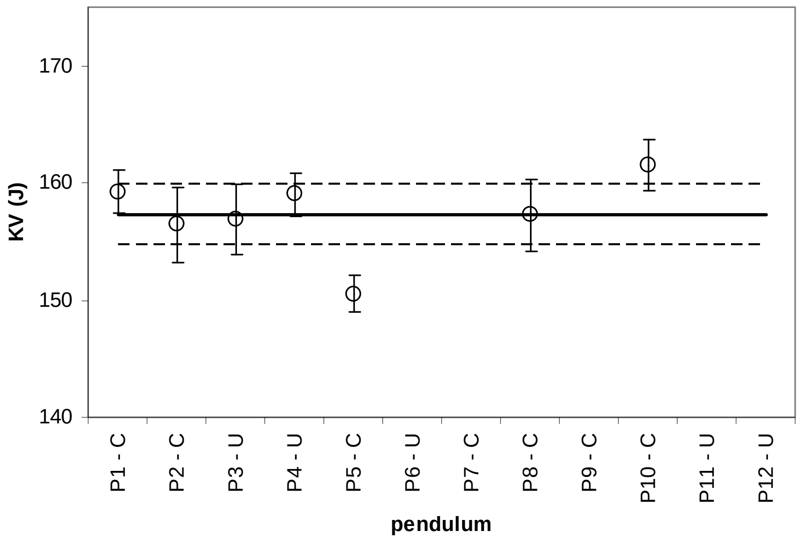

Figure 4: Mean KV values for all 7 accepted data sets. Full line: mean of pendulum mean values; dashed lines: ± 2·u_{char} (u_{char} = standard error of the 7 accepted pendulum mean values). Error bars indicate the pendulum mean value ± 2·s_{within} (s_{within} = standard deviation of the 20 tested test pieces for each pendulum).

Using the single Grubbs' test, it was shown that one of the 7 pendulum mean values is a straggler (95 % confidence level), but none are an outlier (99 % confidence level). The Bartlett test and the Snedecor F-test indicate that the variances of the different pendulums were not homogeneous, therefore pooling of the data between pendulums was not allowed. Further data analysis is based on the pendulum mean values, summarised in Table 3. Skewness and Kurtosis tests indicate that the mean values of the 7 pendulums are normally distributed.

Table 3: Accepted characterisation results: average value, number of successfully tested test pieces, standard deviation, and standard error for each pendulum.

Pendulum code – type	**Average value [J]**	**Number of test pieces**	**Standard deviation [J]**	**Standard error [J]**
P1 – C	159.26	20	4.00	0.90
P2 – C	156.43	20	7.17	1.60
P3 – U	156.84	20	6.73	1.50
P4 – U	159.05	20	4.08	0.91
P5 – C	150.52	20	3.56	0.80
P8 – C	157.25	20	6.75	1.51
P10 – C	161.54	20	4.97	1.11

Earlier observations of a systematic difference between the results obtained with U-type and the C-type hammers [19] are not confirmed. The analysis of the data is therefore done by pooling the pendulum mean values from all 7 pendulums, and the numerical results are summarised in Table 4, which gives the mean of the accepted mean values (KV_{char}), the number of accepted data sets (p), the standard deviation between the accepted mean values (s_{char}), and u_{char}, the resulting uncertainty of KV_{char}. The latter is calculated as $u_{\mathrm{char}} = \frac{s_{\mathrm{char}}}{\sqrt{p}}$. These are the values that are required when later using the master batches in the certification of secondary batches.

Table 4: Summary of the analysis of the results of the ERM®-FA415s characterisation measurements: average value (mean of pendulum means) KV_{char}; number of pendulums contributing accepted data; standard deviation of pendulum mean values; and the resulting uncertainty u_{char}

	Average value KV_{char} [J]	**Number of pendulums p**	**Standard deviation s_{char} [J]**	**Uncertainty of KV_{char} u_{char} [J]**
ERM®-FA415s	157.3	7	3.5	1.4

6.3.3 Analysis of data from instrumented impact testing

The three laboratories which performed instrumented impact tests (on P2, P5 and P9) all reported force-displacement curves with a characteristic oscillation pattern during the loading stage. Figure 5 shows a curve for one test piece, with a shape that is representative for test pieces of both of the 2 batches tested.

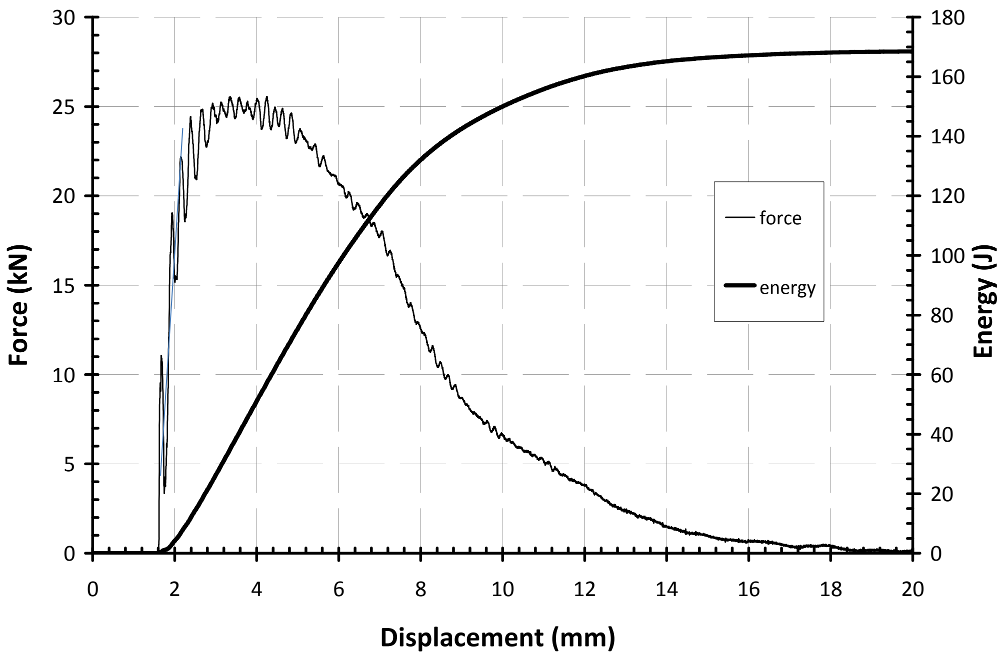

Figure 5: Results of an instrumented Charpy-V test according to ISO 14556 [18]: the measured force, and the absorbed energy (calculated via the integration of the force-displacement signal) versus the displacement of the impacting hammer (calculated via integration of the force-time signal) [image: courtesy J.-L. Puzzolante, SCK-CEN, Mol, BE].

ISO 14556:2000 [18] provides a procedure for determining characteristic values of force, displacement and energy. In particular, the total impact energy (W_t) values were investigated (see Table 5).

Elimination of the results of P9 (as it failed the bias test for non-instrumented results) improved the comparability, but the remaining two results did not provide sufficient basis to draw further firm conclusions. Nevertheless, it is observed that the average results of the two accepted instrumented data sets results match with the average of the non-instrumented values of the same pendulums, as well as with the average of the 7 accepted non-instrumented data sets (overlapping standard deviations).

Table 5: Summary of instrumented impact test results.

Pendulum Code	FA415v		FA415s	
	non-instrumented ($KV \pm s_{\text{within}}$)	instrumented ($W_t \pm s_{\text{within}}$)	non-instrumented ($KV \pm s_{\text{within}}$)	instrumented ($W_t \pm s_{\text{within}}$)
	J	J	J	J
P2	154.2 ± 6.0	156.2 ± 4.9	156.4 ± 7.2	159.5 ± 7.3
P5	151.9 ± 5.3	154.1 ± 5.4	150.5 ± 3.6	152.5 ± 3.6
P9*	171.3 ± 8.2	171.8 ± 8.7	165.8 ± 7.9	165.9 ± 8.3
Average over (P2,P5,P9) ± s between the 3 averages	**159 ± 11**	**161 ± 10**	**158 ± 8**	**159 ± 7**
Average over (P2,P5) ± *s* between the 2 averages	**153.1 ± 1.6**	**155.2 ± 1.5**	**153.5 ± 4.2**	**156.0 ± 4.9**

* Non-instrumented results of P9 were eliminated.

7 Value assignment

ISO Guide 35 [20] provides a generic, ISO Guide 98-3 (GUM) [3] compliant uncertainty model for use in the certification of batches of CRMs. In Charpy terms, the model can be expressed as follows:

$$KV_{\text{MB}} = KV_{\text{char}} + \delta KV_{\text{hom}} + \delta KV_{\text{lts}} + \delta KV_{\text{sts}} \qquad \text{Equation 4}$$

with KV_{char} the KV value obtained from the characterisation of the batch, δKV_{hom} an error term due to variation between test pieces, δKV_{lts} and δKV_{sts} error terms due to the long-term and short-term instability of the CRM. Homogeneity and stability studies are designed in such a way that the values of the corresponding error terms are zero. However, the uncertainties of the error terms are not (always) zero. Assuming independence of the variables, the uncertainty of the certified value of the Charpy CRM can therefore be expressed as:

$$u_{\text{MB}} = \sqrt{u_{\text{char}}^2 + u_{\text{hom,MB}}^2 + u_{\text{lts}}^2 + u_{\text{sts}}^2} \qquad \text{Equation 5}$$

The value of u_{char} is 1.4 J (see Table 4).

With respect to $u_{\text{hom,MB}}$: $u_{\text{hom,MB}}$ (see section **Error! Reference source not found.**) is 5 times smaller than u_{char}. This proves that the differences observed between the 7 pendulum mean values are not due to inhomogeneity of the test pieces, but due to genuine differences in the performance of the different pendulums and in the way they are operated. It can be concluded that the homogeneity contribution to the certified value of a Master Batch is negligibly small. It is repeated here that the test piece-to-test piece heterogeneity of the Master Batch test pieces will be added in the uncertainty budget of the certified value of the secondary batches.

With respect to u_{sts} and u_{lts}: the properties of the steel test pieces are not affected by short term periods of transport. Section 5 explains the decision not to add an uncertainty contribution from instability, and to limit the shelf-life to a period of 10 years.

The certified uncertainty therefore consists only of u_{char}. The effective number of degrees of freedom of the uncertainty value u_{char} is directly calculated as (p - 1), with p the number of accepted data sets. The number of degrees of freedom of u_{MB} (ν_{RM}) is the same as that of u_{char}: ν_{char} = ν_{RM} = 6. The number of degrees of freedom is not sufficiently large to justify the use of a coverage factor k = 2 to expand the confidence level to about 95 %. However, the uncertainty reported on the certificate is the standard uncertainty, with a confidence level of about 68 %, since this is the value that will need to be combined later, during the certification of secondary batches, with other standard uncertainty contributions, with their corresponding degrees of freedom.

8 Metrological traceability

The absorbed energy *KV* is a method-specific quantity, and can only be obtained by following the procedures specified in ISO 148-1 [2]. The certified value of the new master batch certified in this study is defined by these standard procedures as it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures.

The certified value of the new master batch certified in this study is traceable to the SI, since the results were obtained on pendulums regularly verified using tools that are calibrated in an SI-traceable manner.

9 Commutability

The commutability issue concerns both the choice of material as well as the method chosen to characterise the reference material.

During this certification study, 12 different pendulums were used, each equipped with an ISO-type striker of 2 mm striker edge radius [2]. The reference materials are commutable if tested with 2 mm strikers, and when following the ISO standard test procedures [1,2]. The certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm striker edge radius [4].

The steel chosen is of an industrial type, combining hardness and absorbed energy properties that impose forces on the pendulum that cover the same range of forces as met in routine use. The reference material is therefore bound to trigger the same potential instrumental problems as those that are experienced in practice. This guarantees the commutability of the reference material.

10 Summary of results

The certified value and associated uncertainty are summarized in Table 6.

Table 6: Certified value and associated uncertainty for ERM®-FA415s.

STEEL CHARPY V-NOTCH TEST PIECES		
	Impact toughness	
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	157.3	1.4

1) The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in ISO 148-1 [2]. The certified value is only valid for strikers with a 2 mm tip radius and in the temperature range of (20 ± 2)°C.

2) The certified value is estimated as the mean of means of absorbed energies measured on 7 pendulums at 5 different laboratories. On each pendulum, 20 test pieces were broken. The pendulums used are regularly verified with equipment that is calibrated in a manner that is traceable to the International System of Units (SI). Therefore, the certified value is traceable to the International System of Units (SI).

3) Standard uncertainty *u* of the certified mean absorbed energy of batch ERM-FA415s, estimated as the standard error of the mean of the 7 pendulum mean values, corresponding with a confidence level of about 68 %, as defined in the ISO/IEC Guide 98-3:2008; Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM) [3]. The number of degrees of freedom of the certified uncertainty, $\nu_{RM} = 6$.

11 Instructions for use

11.1 Intended use

Test pieces of ERM®-FA415s correspond to the '(certified) BCR test pieces' as referred to in the (now obsolete) EN 10045-2 [21], as well as to the 'certified reference test pieces' as defined in ISO 148-3 [5]. In particular, the test pieces of this batch are intended for use by IRMM in the certification of secondary batches of certified reference test pieces for the indirect verification of impact testing machines with a striker of 2 mm edge radius according to procedures described in detail in ISO 148-2 [1].

11.2 Test piece preparation

Special attention is drawn to the cleaning and conditioning of the specimens prior to testing. It is mandatory to remove the oil from the test piece surface prior to testing, without damaging the edges of the test piece. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the test piece clean.

The following procedure is considered good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the test piece, but do not use hard (e.g. steel) brushes to remove the oil from the notch.

2. Submerge the test pieces in ethanol for about 5 minutes. Use of ultrasonication is encouraged, but only if the edges of the test pieces are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once the test pieces are removed from the solvent, only manipulate the test pieces wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

11.3 Pendulum impact tests

After cleaning, the test pieces need to be broken with a pendulum impact test machine in accordance with ISO 148-2 [1] , including the correction of the raw data for friction. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed.

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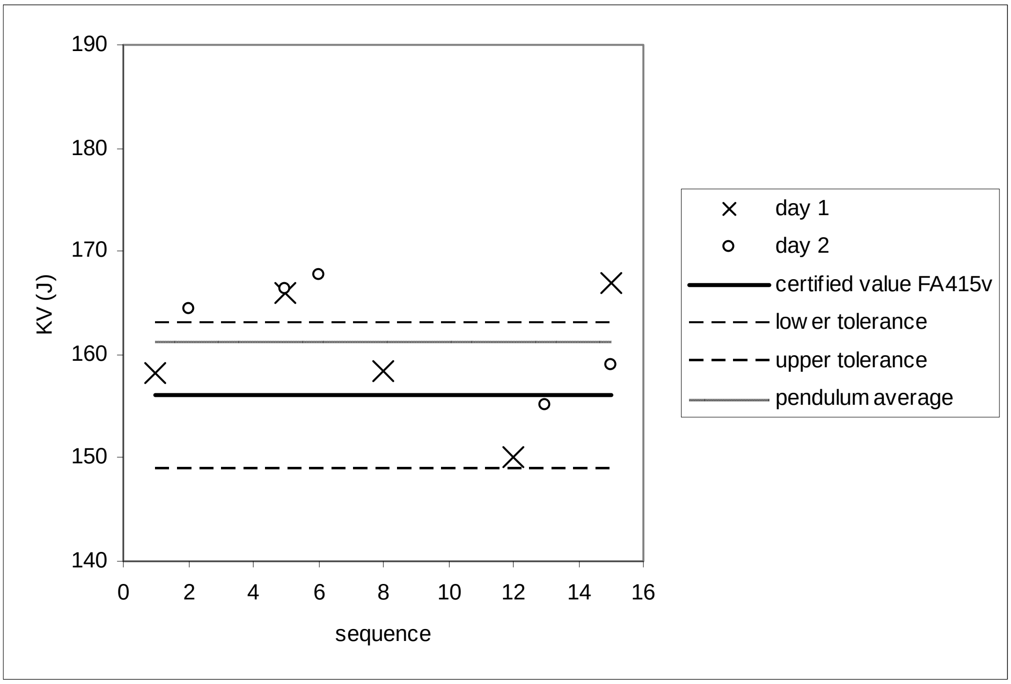
Annex 1: Details of pendulums in characterisation laboratories

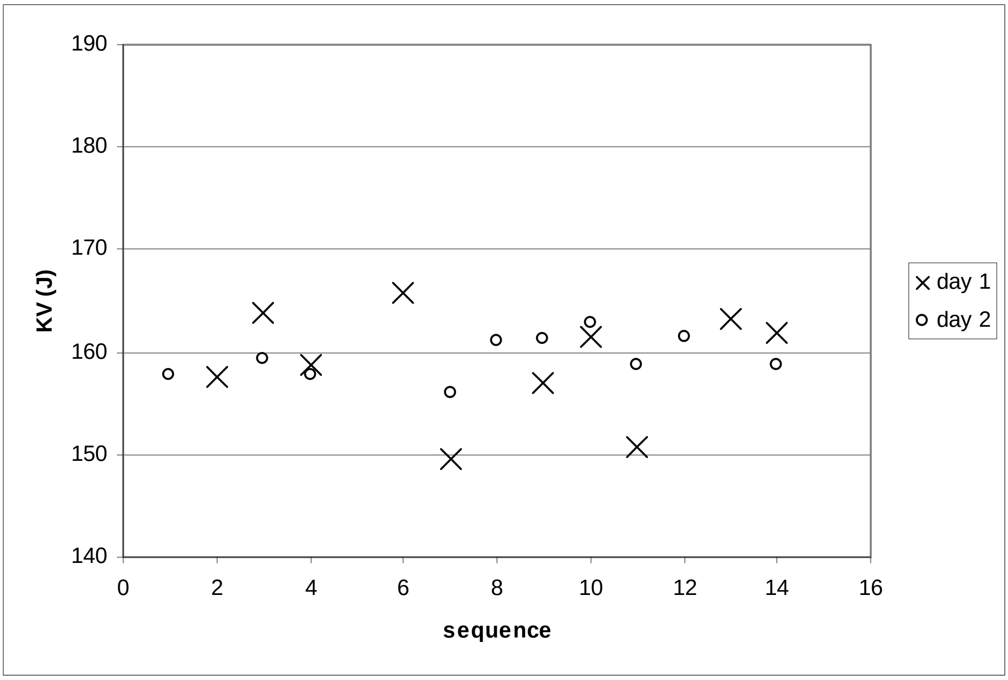
Pendulum code	Constructor / type	Hammer type	Nominal energy	Pendulum moment
			J	Nm
P1	Wolpert PW30	C	300	152.7
P2	Amsler Otto Wolpert PW30	C	300	154.2
P3	Satec SI-3	U	409	237.4
P4	Tinius Olsen 74 Impact	U	359	239.5
P5	Mohr & Federhaff PSW 30/15	C	300	151.5
P6	Roell & Korthaus RKP300	U	300	161.5
P7	MFL PSW300	C	300	154.1
P8	Tokyo Koki	C	356	265.5
P9	Toni-MFL PSW300	C	300	154.1
P10	Instron Wolpert PW-30	C	300	155.8
P11	Amsler Otto Wolpert PW 30/15	U	300	153.9
P12	Zwick RKP450A	U	300	160.2

Annex 2: Individual data of characterisation laboratories

P1: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

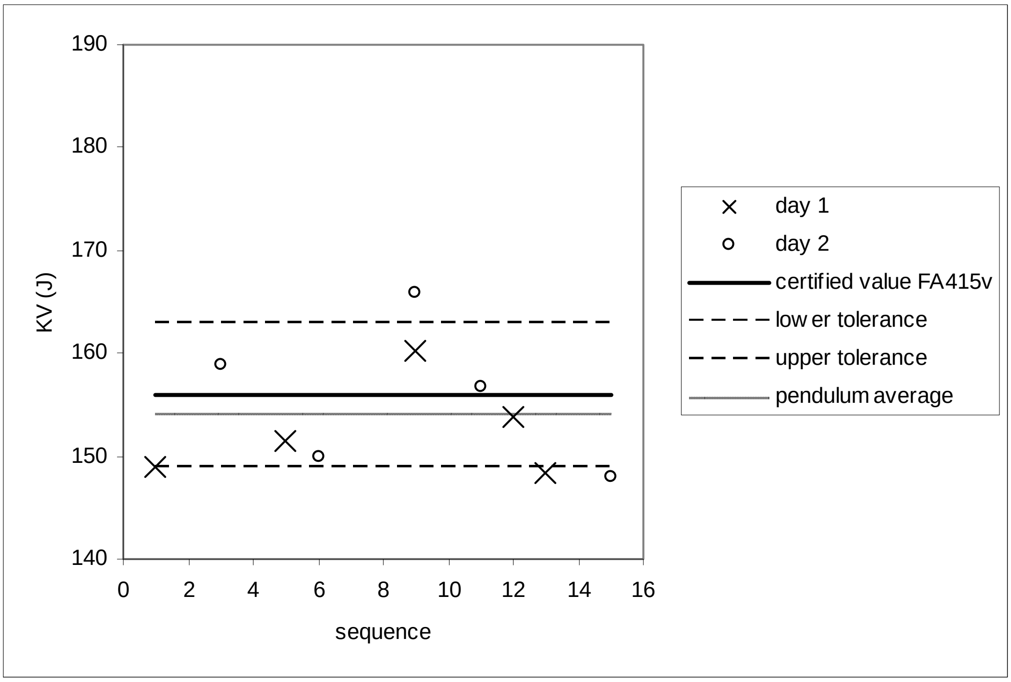


a) results obtained on FA415v test pieces

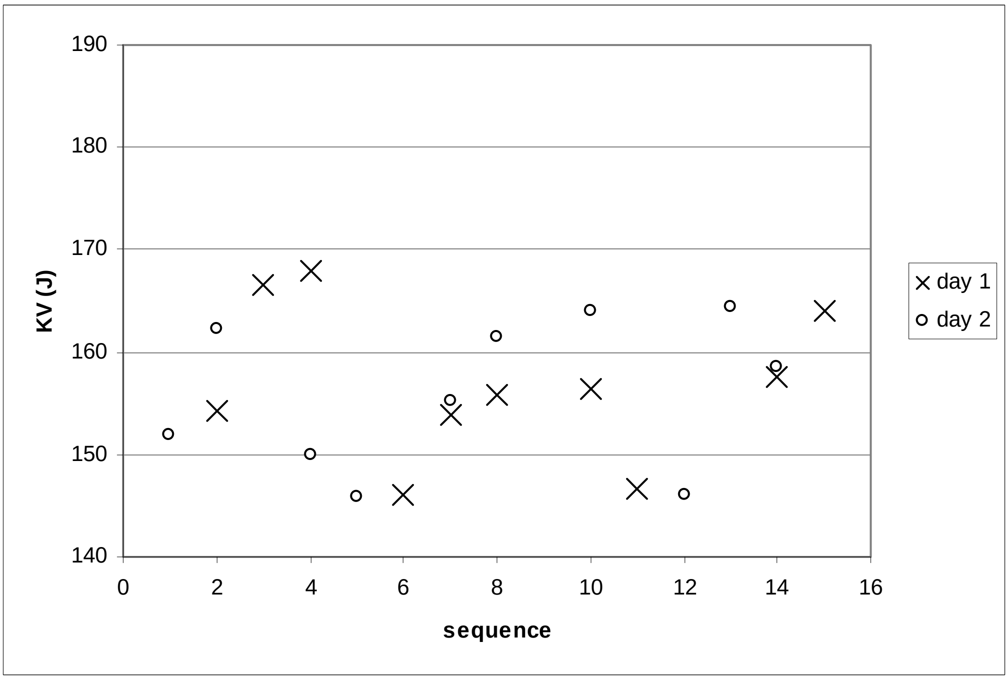


b) results obtained on FA415s test pieces

P2: *KV KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

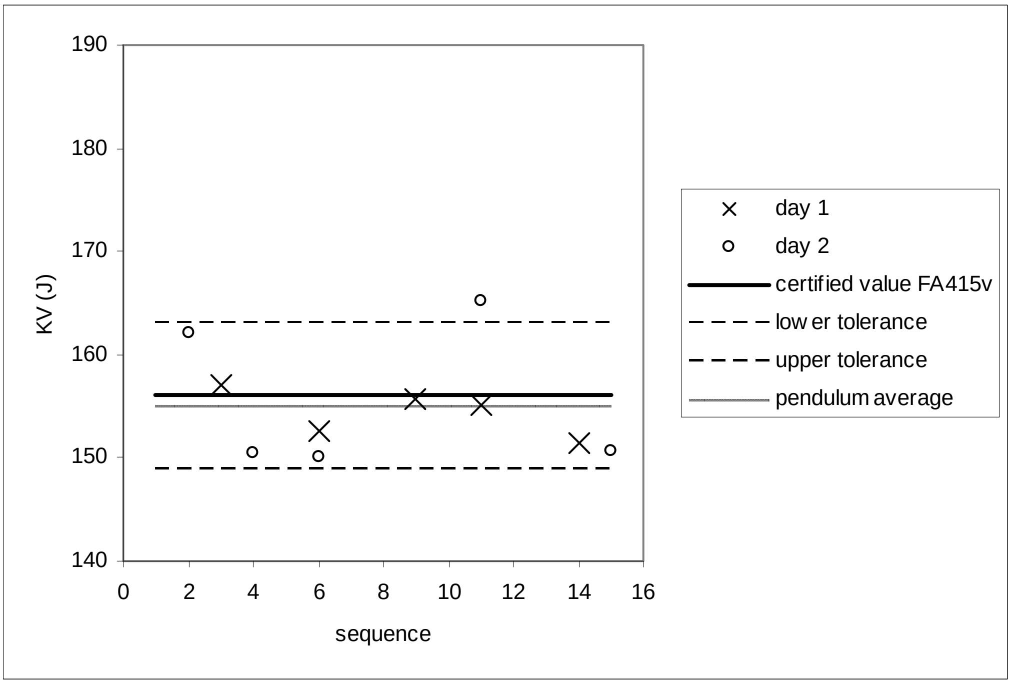


a) results obtained on FA415v test pieces

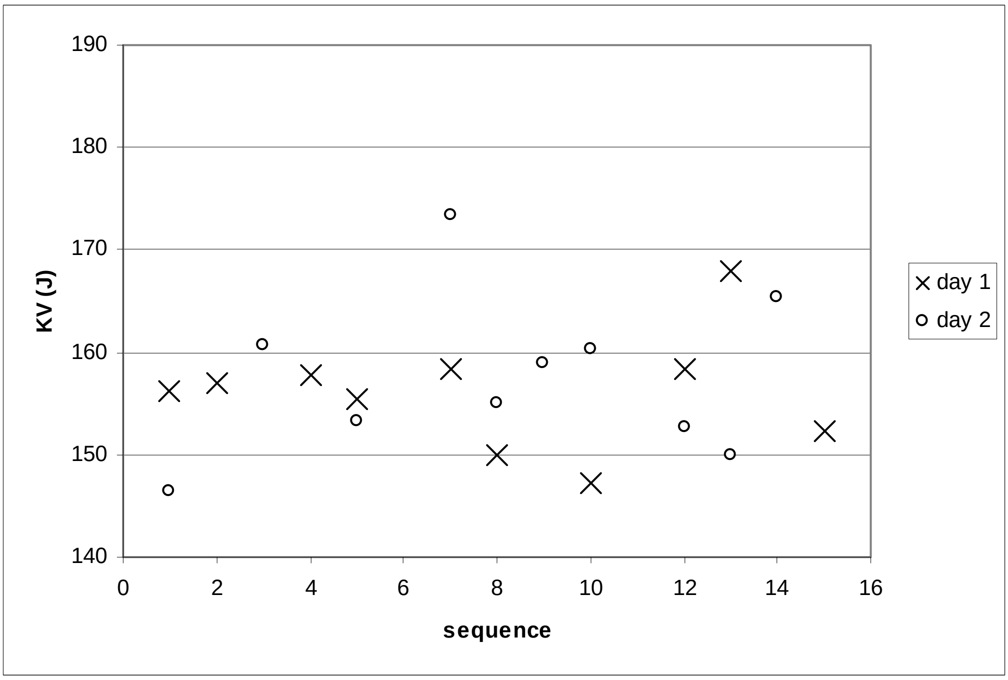


b) results obtained on FA415s test pieces

P3: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

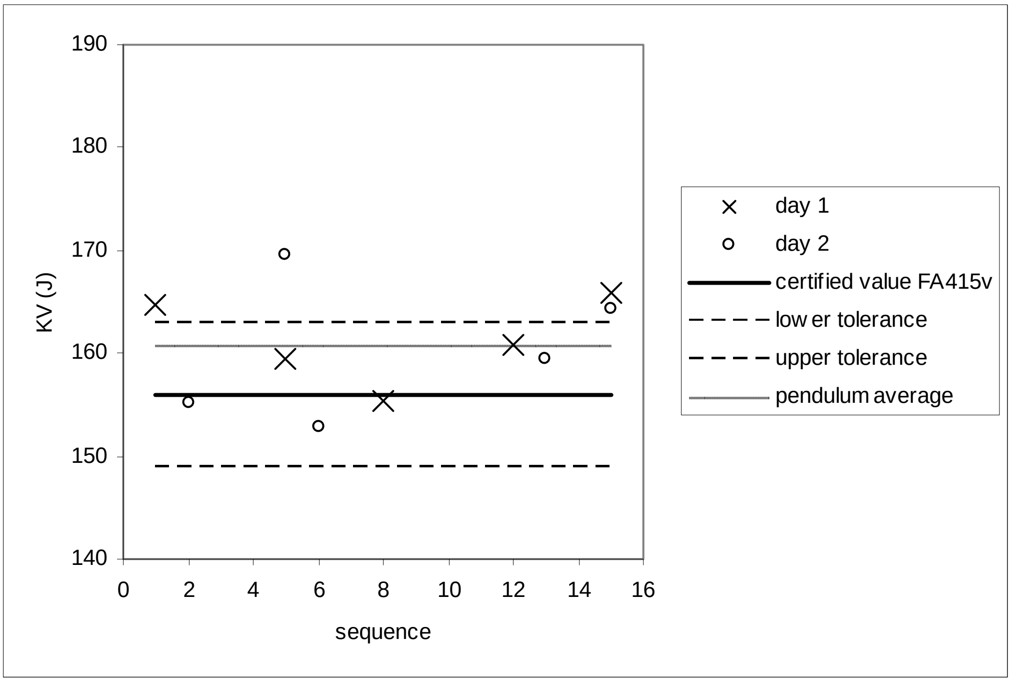


a) results obtained on FA415v test pieces

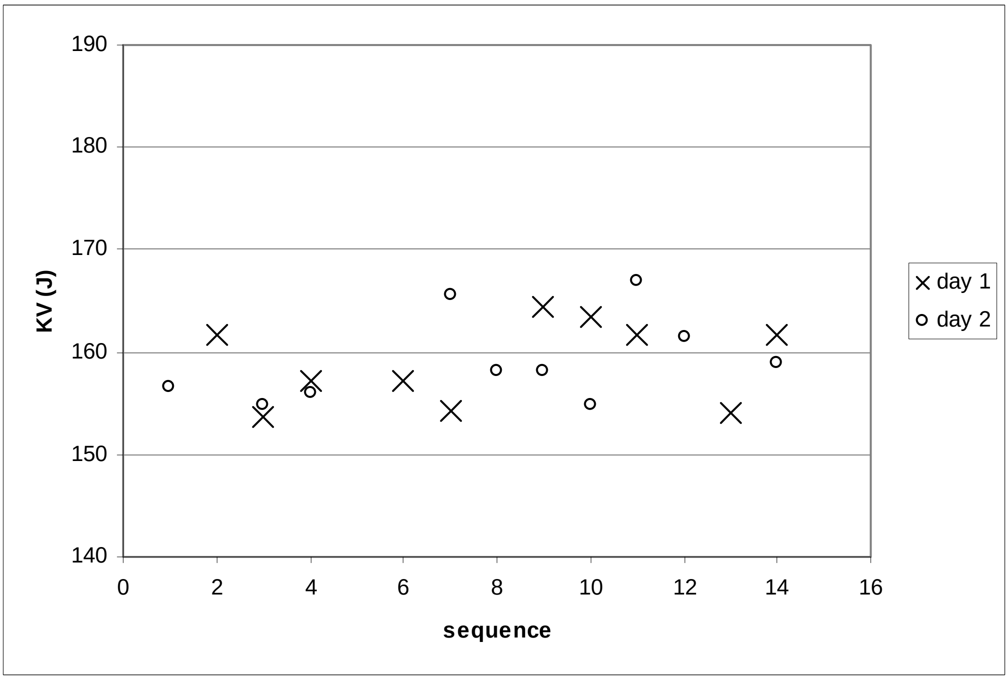


b) results obtained on FA415s test pieces

P4: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

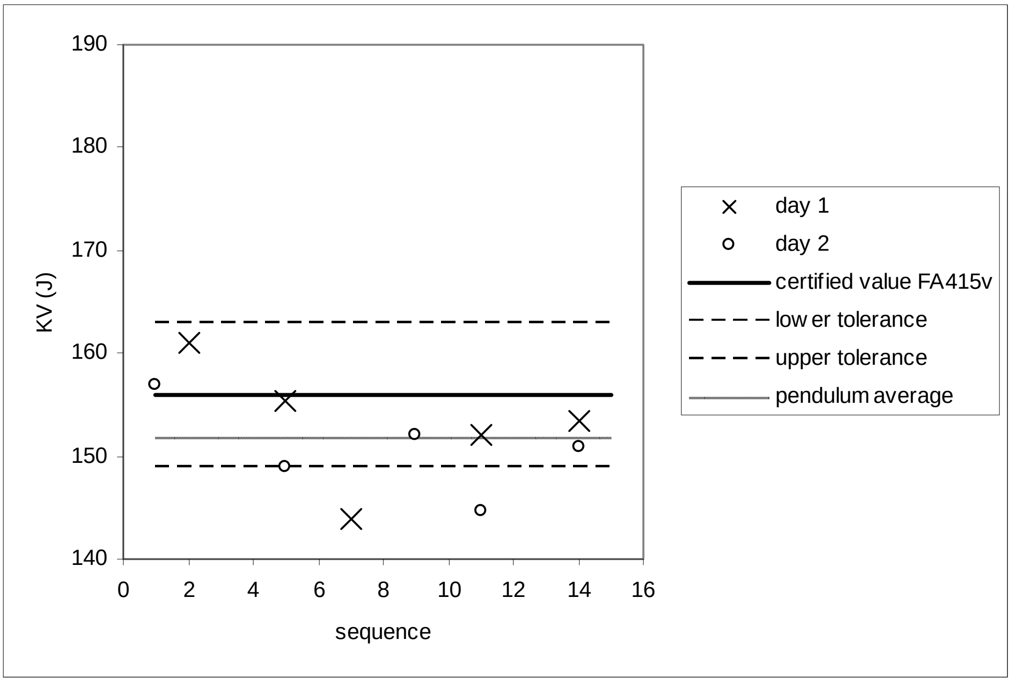


a) results obtained on FA415v test pieces

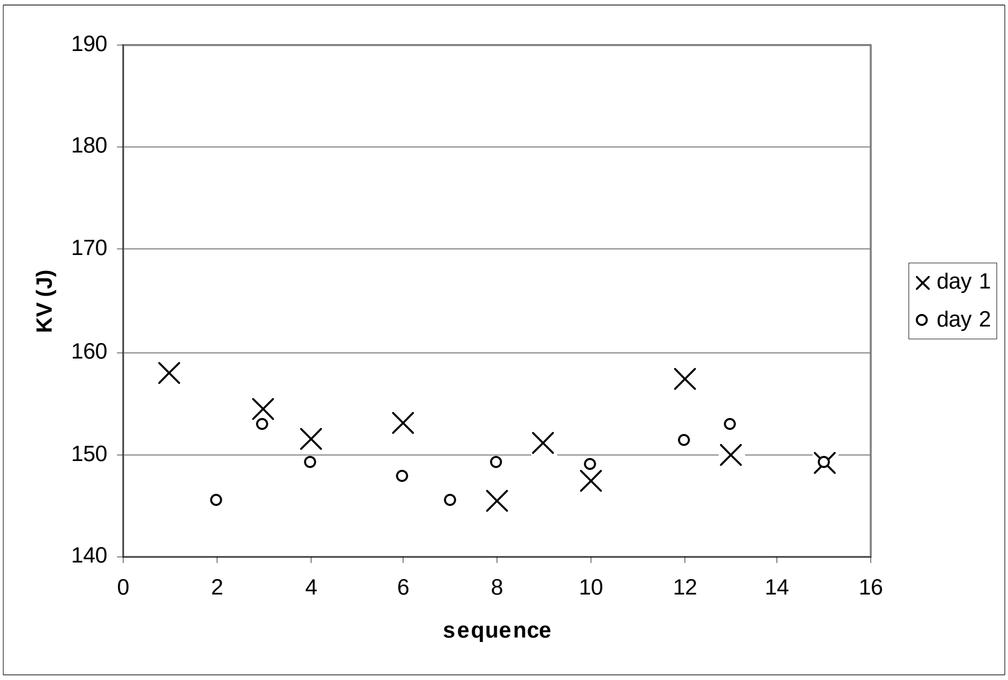


b) results obtained on FA415s test pieces

P5: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

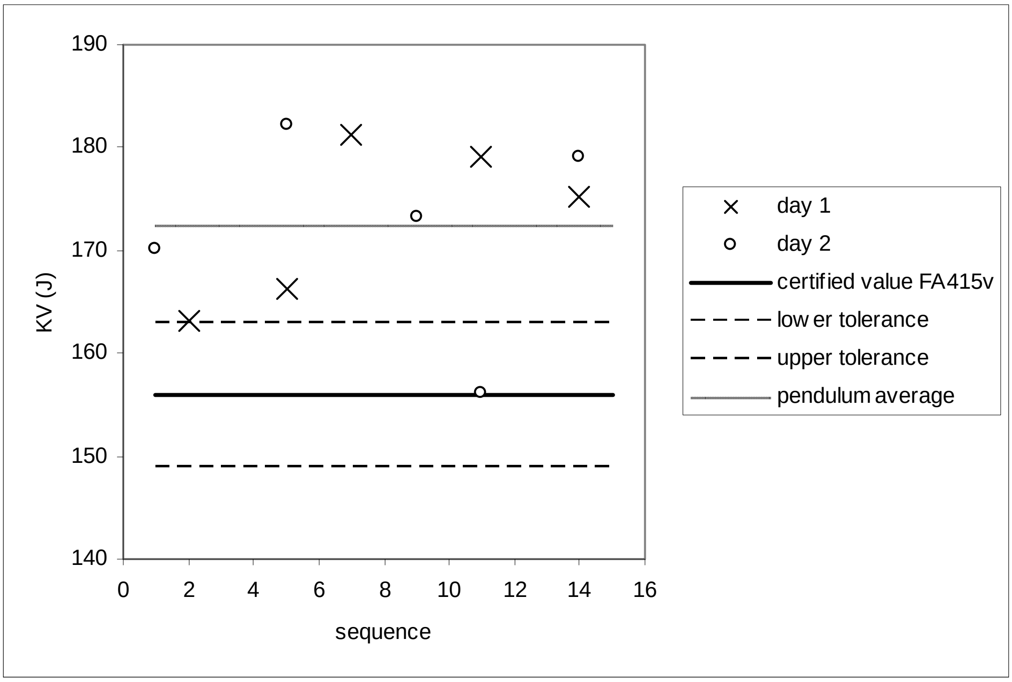


a) results obtained on FA415v test pieces

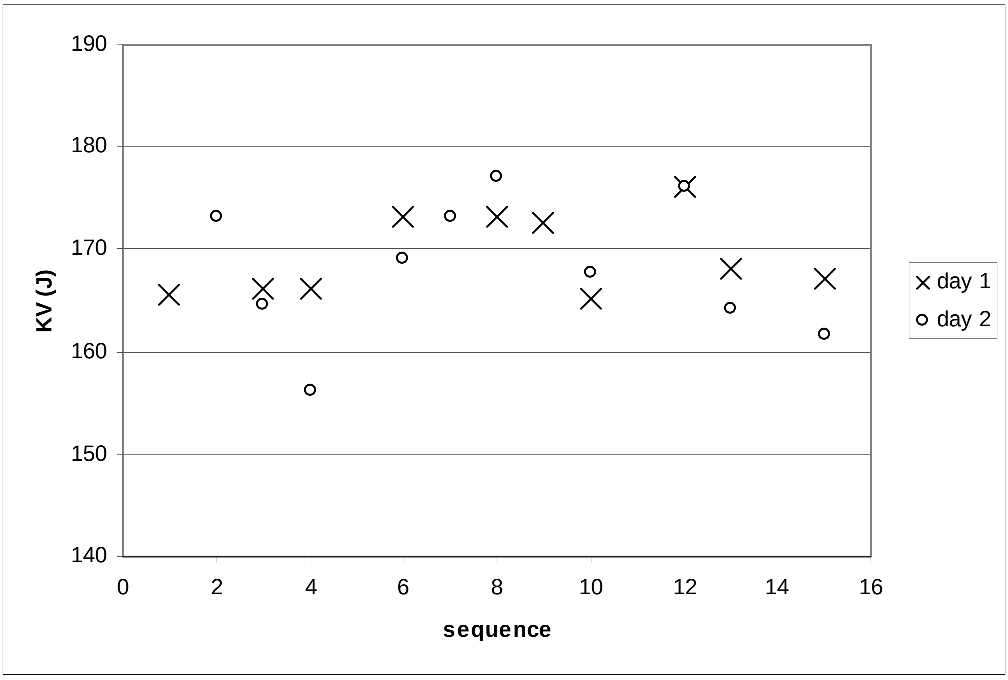


b) results obtained on FA415s test pieces

P6: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

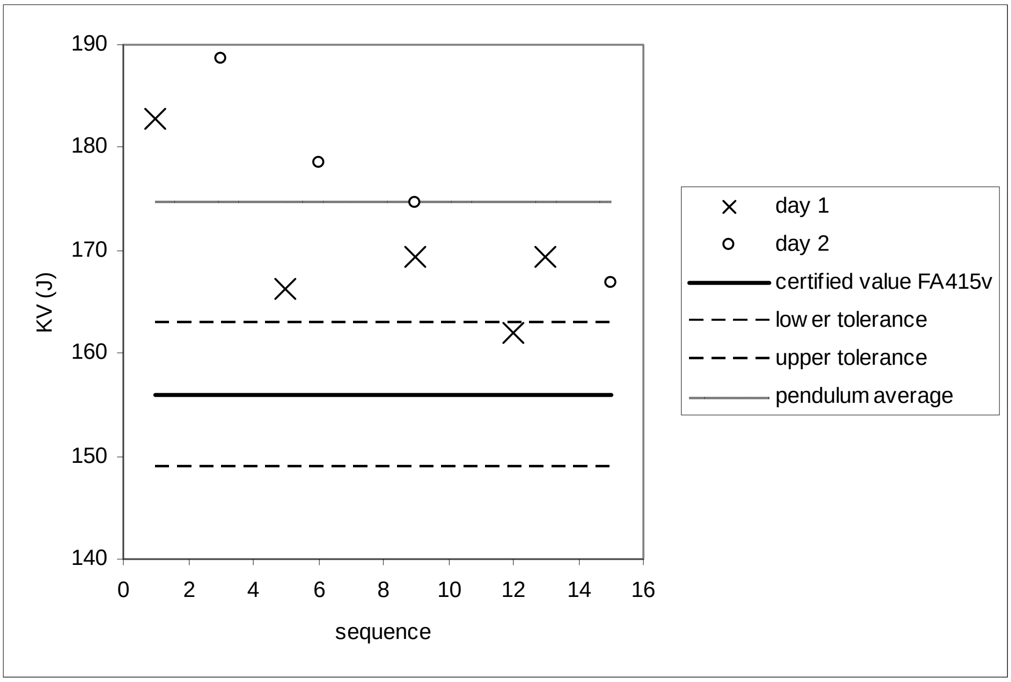


a) results obtained on FA415v test pieces

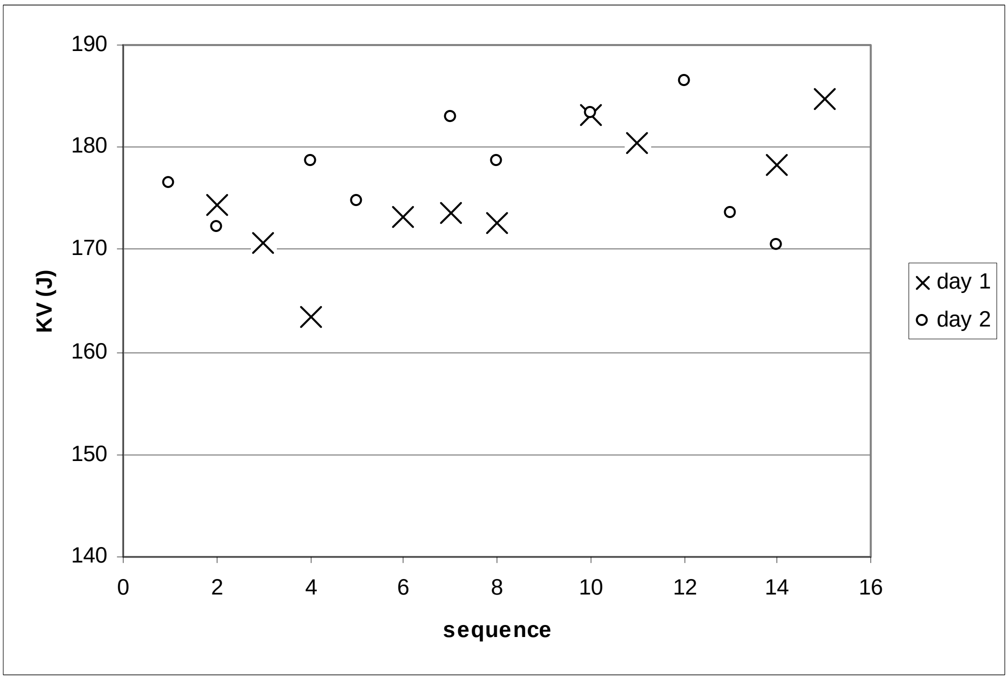


b) results obtained on FA415s test pieces

P7: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

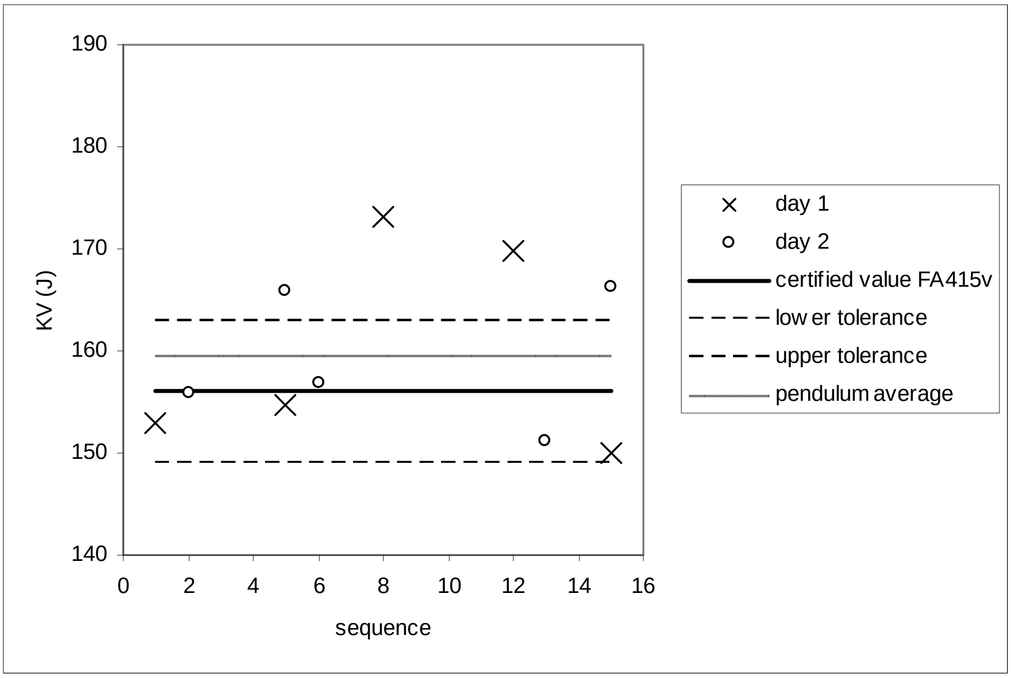


a) results obtained on FA415v test pieces

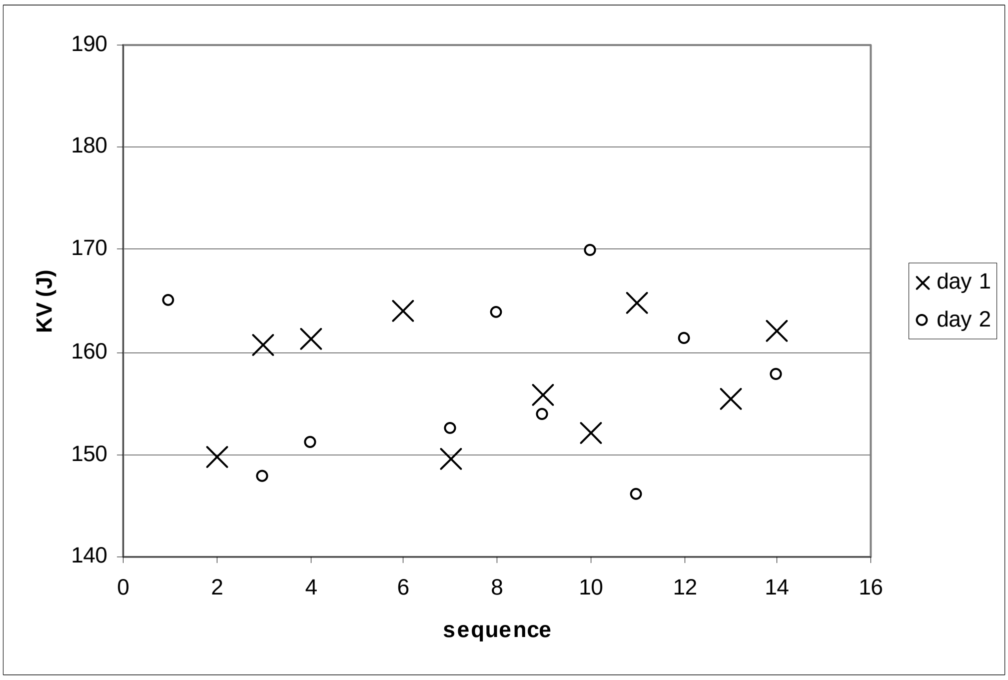


b) results obtained on FA415s test pieces

P8: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

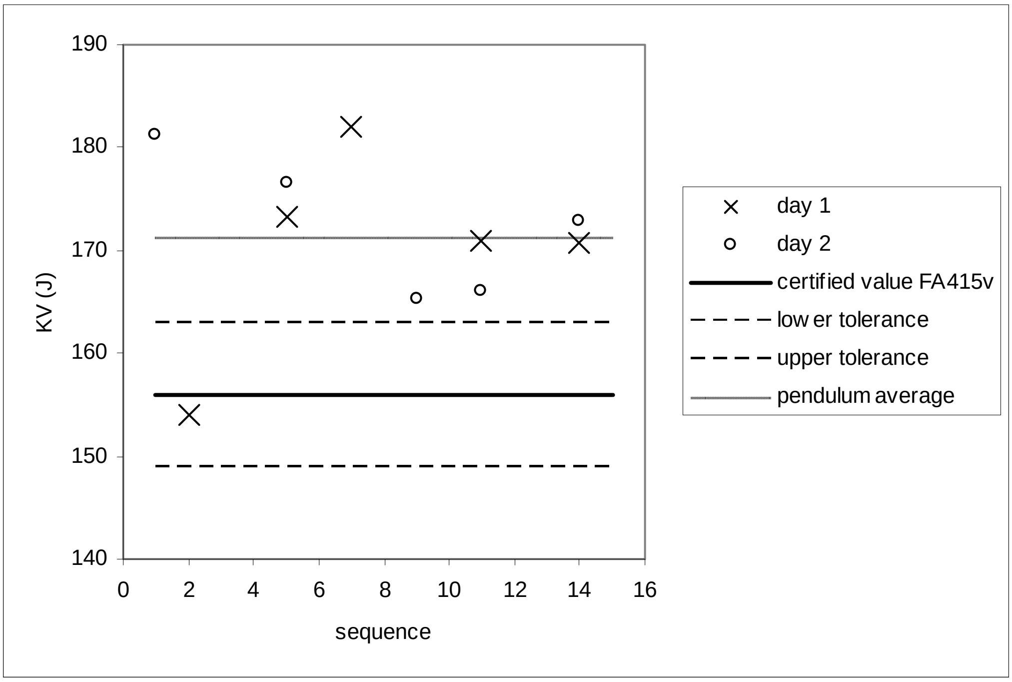


a) results obtained on FA415v test pieces

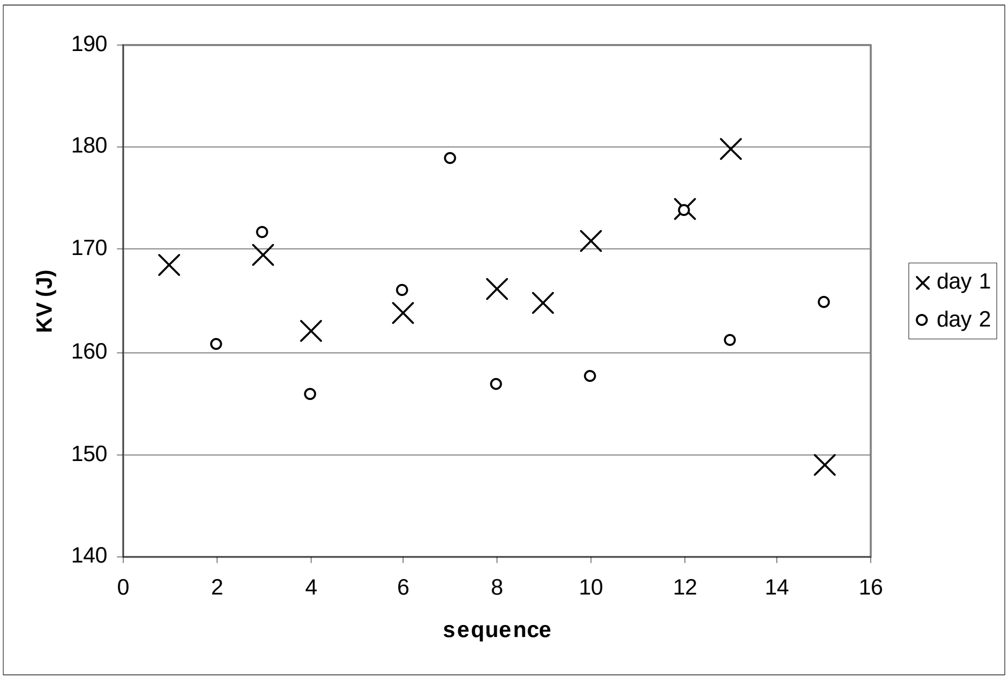


b) results obtained on FA415s test pieces

P9: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

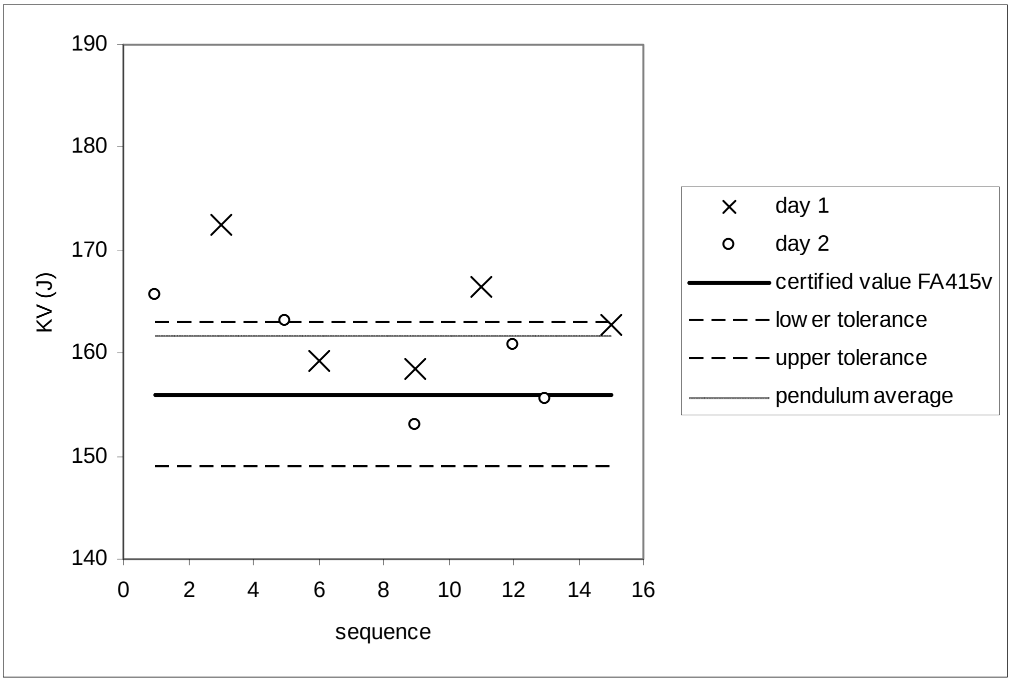


a) results obtained on FA415v test pieces

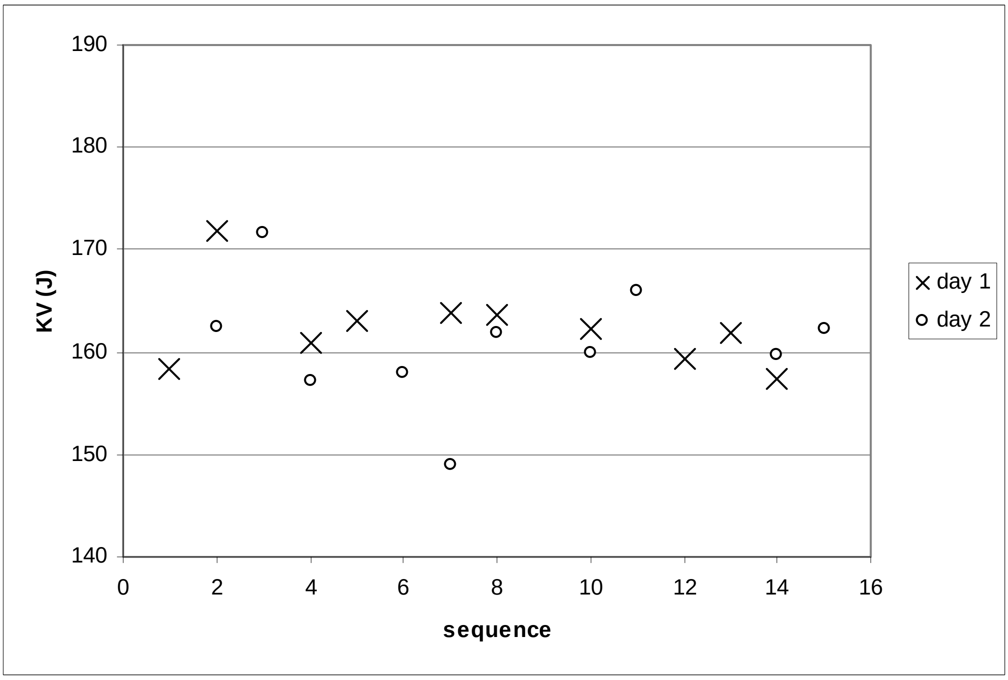


b) results obtained on FA415s test pieces

P10: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

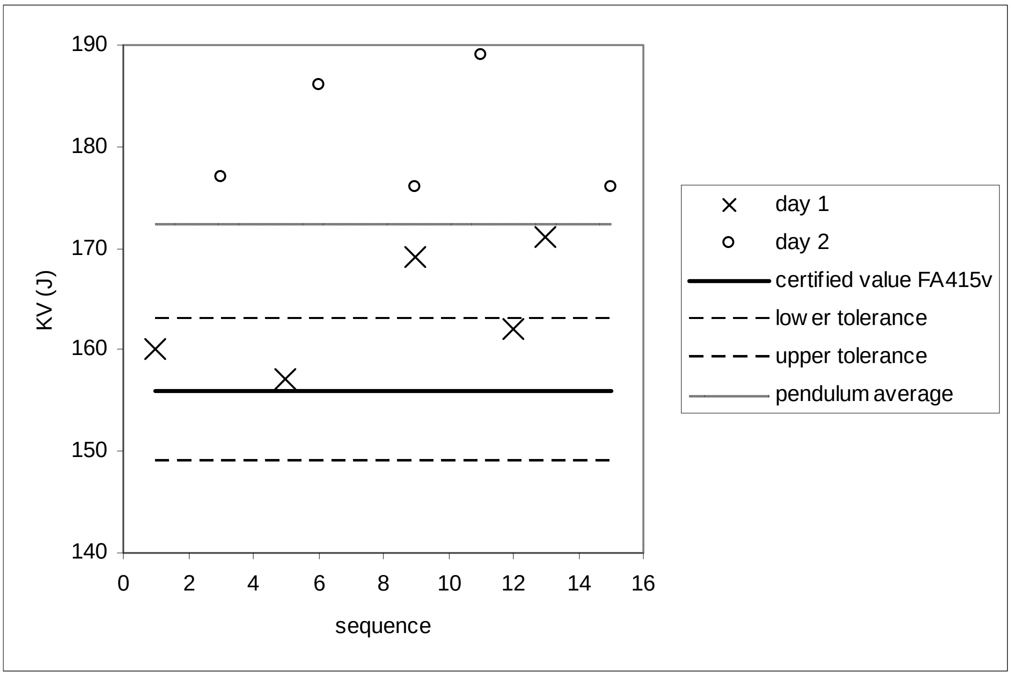


a) results obtained on FA415v test pieces

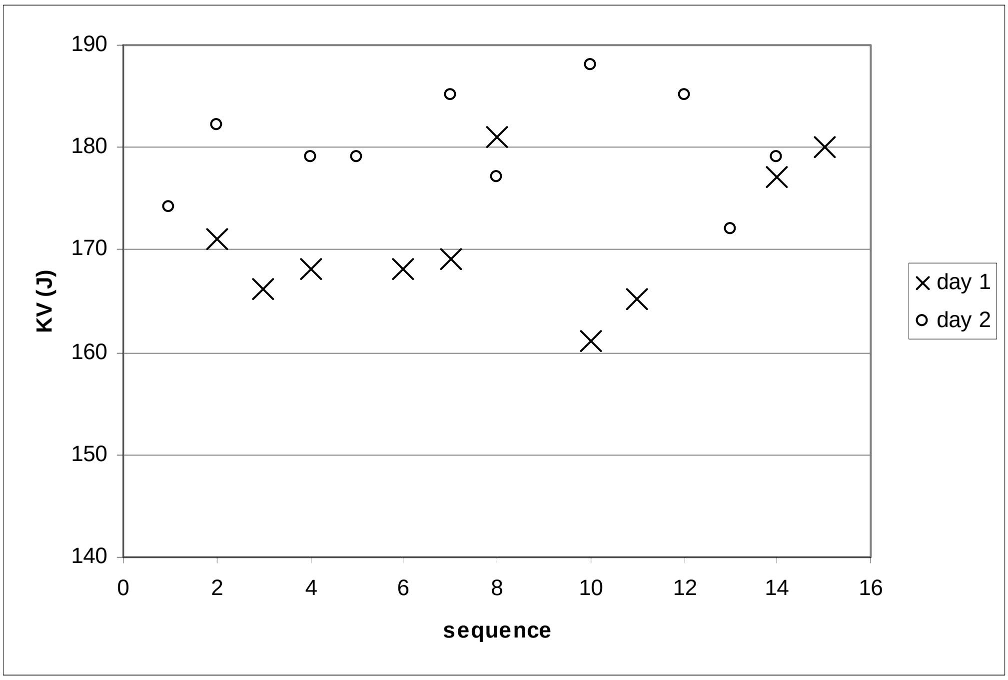


b) results obtained on FA415s test pieces

P11: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.

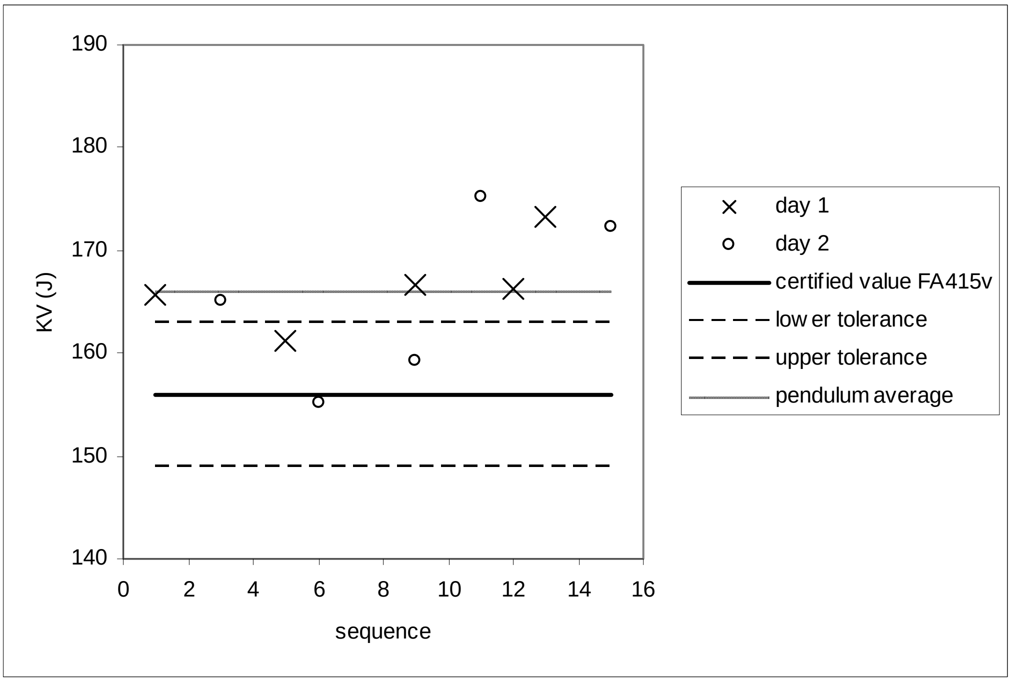


a) results obtained on FA415v test pieces

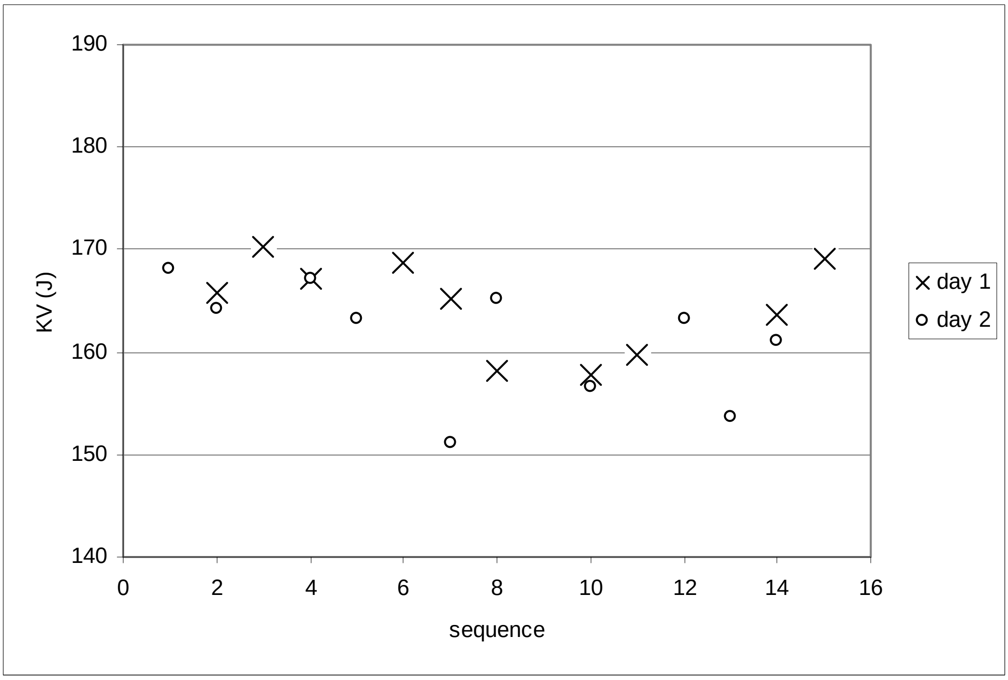


b) results obtained on FA415s test pieces

P12: *KV* data versus test sequence; a) Comparison of data obtained on FA415v test pieces with the certified value and the ISO 148-3 verification tolerances; b) Results obtained on FA415s test pieces.



a) results obtained on FA415v test pieces



b) results obtained on FA415s test pieces

European Commission

EUR 25380 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: The certification of absorbed energy (150 J nominal) of a Master Batch of Charpy V-notch reference test pieces:ERM®-FA415s
Author(s): G. Roebben, A. Dean, A. Lamberty
European Commission, Joint Research Centre Institute for Reference Materials and Measurements (IRMM) Geel, Belgium
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Abstract
This certification report describes the certification of ERM®-FA415s, a batch of steel Charpy V-notch certified reference test pieces. This batch will serve as a Master Batch, to be used by IRMM for the certification of secondary batches. Sets of five pieces taken from a secondary batch are distributed by IRMM and its authorised distributors for the verification of pendulum impact test machines according to ISO 148-2 [1].

The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1 [2]. The certified value for *KV* is an estimate of the mean value of the whole batch. The obtained values are shown in the table below. The associated uncertainties are standard uncertainties corresponding to a confidence level of about 68 %. The certified value is traceable to the International System of Units (SI). The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

LA-NA-25380-EN-N

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